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(54) PROCESS FOR THE MANUFACTURE OF POLYETHYLENE TEREPHTHALATE GRANULES

(71) We, FARBWERKE HOECHST AKTIENGESELLSCHAFT, vormals Meister Lucius & Brüning, a Body Corporate recognised under German Law, of 6230 Frankfurt (M)-Hoechst, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement: —

The present invention relates to a process for the manufacture of polyethylene terephthalate granules which, after exposure to a thermal treatment in the solid phase, may be worked up by injection-moulding.

Polyethylene terephthalate acquires outstanding mechanical properties after exposure to a thermal treatment in the solid phase at temperatures above 200°C owing to the resulting condensation to higher molecular weights and this plastics material in the highly condensed form can be worked up by injection - moulding. However, when being worked up in injection machines, the unprocessed plastics material has to meet certain standards of shape. It must, for example, be free flowing, it must neither stick nor have a tendency to bridging since otherwise the uniform feeding of the plastics material into the injection machine is not assured. The conventional granulating processes applied to polyethylene glycol terephthalate, for example continuous granulation, usually yield granular products, so-called chips, which are not suitable for being worked up in injection machines. Polyesters having a specific viscosity of approximately 800, which are used as basic materials for the thermal condensation in the solid phase, can be extruded by means of the conventional extruders with only great difficulty, for example into strand-shaped granules. A polyester having a specific viscosity of, for example, 1400 or 1500, which has undergone a thermal treatment, has a better behaviour in this respect. It can be worked up without difficulty into strand-shaped granules so that it is easy, after a thermal treatment has been carried out, to obtain the desired granular shape. However, this method requires the additional expensive process steps of re-melting and extruding and results in moreover, a partial degradation of the polyethylene terephthalate whereby the result of the thermal treatment is partially rendered ineffective.

The present invention provides a process for the manufacture of polyethylene terephthalate granules suitable for injection moulding, which process comprises transforming a polyethylene terephthalate melt into granules by means of an underwater granulator, and subjecting these granules to thermal treatment in the solid phase at a temperature greater than 200°C.

The basic material to be granulated is obtained by a conventional melt condensation and generally has a specific viscosity within the range of from 700 to 1000, preferably of from 800 to 850. The polyester melt, which preferably has a temperature of approximately 280°C., is conducted to an underwater granulator being designed, for example, such that the hot strand of plastics material extruded from the die is seized by a water jet issuing from a tubular die, cooled, and passed through an underwater cooling zone to a cutting device. The length and the cross-section of the cylindrical granules formed can be controlled by the speed of the cutting device and by the draw-off speed of the water. The wet granular product is passed over a draining screen, and then dried.

The granules obtained by the above granulation process each have, for example, the shape of a cylinder or a slightly flattened cylinder, with a length of from 3 to 4 mm and a diameter of from 2.5 to 3 mm. The special advantage of the above granulation process is that a granular product of a very uniform shape is obtained without dust and cuttings, and this product is extremely suitable for a subsequent thermal condensation in the solid phase at a temperature above

200°C. to form high molecular weight polyesters having a molecular weight of about 15000 to 25000 and a specific viscosity of about 1400 to 1500. The polyesters so obtained are particularly suitable for being worked up by injection moulding.

EXAMPLE

From an agitator vessel, in which 1.6 tons of polyethylene terephthalate melt had been highly condensed *in vacuo* to a specific viscosity of approximately 800, the melt, which had a temperature of 280°C., was forced out by means of a nitrogen pressure of 10 atmospheres gauge during the course of approximately 45 minutes, through an underwater granulator installed at the bottom of the vessel, said granulator consisting of 36 die holes and the same number of water jets. Each die hole had a circular opening of a diameter of 6 mm; the total throughput of water for taking away and cooling the strand amounted to 150 cu.m./h. The issuing strands, after having passed through a cooling zone of approximately 4 m length, were cut up by a cutting device whose speed was adjusted such that a granular product of a length of 3 mm was obtained. The diameter of the cylindrical granules was 2.5 to 3 mm. On issuing from the cutting device, the granules were passed over a screen to separate the solid material from the water, the latter being recirculated *via* a condenser. The wet granular product was, after drying, subjected to a thermal post-condensation.

WHAT WE CLAIM IS:—

1. A process for the manufacture of polyethylene terephthalate granules suitable for injection moulding, which process comprises transforming a polyethylene terephthalate melt into granules by means of an underwater granulator, and subjecting these granules to thermal treatment in the solid phase at a temperature greater than 200°C.

2. A process as claimed in claim 1, wherein the polyethylene terephthalate melt has a specific viscosity within the range of from 700 to 1000.

3. A process as claimed in claim 2, wherein the specific viscosity is from 800 to 850.

4. A process as claimed in any one of claims 1 to 3, wherein the polyethylene terephthalate melt has a temperature of approximately 280°C.

5. A process as claimed in any one of claims 1 to 4, wherein each of the granules (before thermal treatment) has the shape of a cylinder or a slightly flattened cylinder with a length of from 3 to 4 mm and a diameter of from 2.5 to 3 mm.

6. A process as claimed in claim 1, conducted substantially as herein described.

7. Polyethylene terephthalate granules whenever prepared and treated by a process as claimed in any one of claims 1 to 6.

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